

Study on Solution Properties of Binary Mixtures of Some Industrially Important Solvents with Cyclohexylamine and Cyclohexanone at 298.15 K

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Received: 16 September 2009 / Accepted: 7 May 2010 / Published online: 30 May 2010
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Abstract Densities and viscosities were measured for the binary mixtures of cyclohexylamine and cyclohexanone with butyl acetate, butanone, butylamine, *tert*-butylamine, and 2-butoxyethanol at 298.15 K over the entire composition range. From density data, the values of the excess molar volume (V^E) have been calculated. The experimental viscosity data were correlated by means of the equation of Grunberg–Nissan. The density and viscosity data have been analyzed in terms of some semiempirical viscosity models. The results are discussed in terms of molecular interactions and structural effects. The excess molar volume is found to be either negative or positive depending on the molecular interactions and the nature of the liquid mixtures and is discussed in terms of molecular interactions and structural changes.

Keywords Density · Excess molar volume · Molecular interaction · Structural effects · Viscosity

1 Introduction

The density and viscosity of pure chemicals and their binary mixtures over the whole composition range at a particular temperature are useful for a full understanding of their thermodynamic and transport properties as well as for practical chemical engineering purposes. Excess thermodynamic properties of solvent mixtures correspond to the

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difference between the actual property and the property if the system behaves ideally and, thus, are useful in the study of molecular interactions and structural arrangements. The present work is a continuation of our systematic study [1,2] of the physicochemical properties of nonaqueous binary liquid mixtures, and this article reports densities (ρ), viscosities (η), and ultrasonic speeds (u) for binary mixtures of cyclohexylamine + (butylacetate, butanone, butylamine, *tert*-butylamine, and 2-butoxyethanol) and cyclohexanone + (butylacetate, butanone, butylamine, *tert*-butylamine, and 2-butoxyethanol) over the entire range of composition at 298.15 K and at atmospheric pressure. Cyclohexylamine is important in characterizing the associated nature of the liquids in mixtures, because of the ability of both a proton donor and a proton acceptor, and these mixtures form water insoluble compounds of medical importance [3]. It is well known that cyclohexanone has numerous scientific and industrial applications. Also, 2-butoxyethanols have wide use as monomers in the production of polymers and emulsion formulations. They are also of considerable interest for studying the heteroproximity effects of etheric oxygen on the –O–H bond and, hence, their influence on the associated nature of the species in these molecules. Properties of mixtures are useful for designing many types of transport and process equipment in the chemical industry [4–8].

2 Experimental

2.1 Chemicals

Cyclohexylamine (Merck, India), cyclohexanone, butyl acetate, butylamine, *tert*-butylamine, and butanone (S.D. Fine Chemicals, Analytical Reagent, purity >99%) were used without further purification. 2-Butoxyethanol (S.D. Fine Chemicals, AR, India) was purified as described in the literature [9]. The pure chemicals were stored over activated 4 Å molecular sieves to reduce water content before use. The chemicals after purification were 99.8% pure, and their purity was ascertained by GLC and also by comparing experimental values of densities and viscosities with those reported in the literature [2, 10–13], when available, as presented in Table 1.

Table 1 Comparison of experimental densities ρ , viscosities η , and sound velocities u , of the pure solvents with literature data at 298.15 K

Solvent	$\rho \times 10^{-3}$ (kg · m ⁻³)		η (mPa · s)		u (m · s ⁻¹)	
	Expt.	Lit.	Expt.	Lit.	Expt.	Lit.
Cyclohexylamine	0.86230	0.8668 [11]	1.7531	1.753 [11]	1416.4	1416.4 [11]
Cyclohexanone	0.94161	0.9416 [12]	2.0198	2.020 [12]	1412.0	
Butylacetate	0.87441	0.8744 [2]	0.6682	0.668 [2]	1176.0	
2-Butanone	0.79812	0.7981 [2]	0.4000	0.378 [10]	1195.0	1195.4 [11]
Butylamine	0.73189	0.73190 [2]	0.4932	0.493 [2]	1251.6	
<i>tert</i> -Butylamine	0.69603	0.6860 [13]	0.4391	0.439 [13]	1260.0	
2-Butoxyethanol	0.90202	0.9020 [13]	2.7823	2.782 [11]	1305.6	1303.2 [11]

2.2 Apparatus and Procedure

The densities were measured with an Ostwald–Sprengel type pycnometer having a bulb volume of 25 cm³ and an internal diameter of the capillary of about 0.1 cm, calibrated at 298.15 K, 308.15 K, and 318.15 K with double-distilled water and benzene [1, 4, 7]. The pycnometer with the test solution was equilibrated in a thermostatic water bath maintained at ± 0.01 K of the desired temperature. Averages of triplicate measurements were taken into account. The reproducibility in mole fraction was within ± 0.0002 . The mass measurements, accurate to 0.01 mg, were made on a digital electronic analytical balance (Mettler, AG 285, Switzerland). The total uncertainty of the density is 3×10^{-4} g · cm⁻³, and that of the temperature is 0.01 K. The viscosity was measured by means of a suspended Ubbelohde type viscometer [14], which was calibrated at 298.15 K with triple-distilled water and purified methanol using density and viscosity values from the literature [7, 11, 15]. The flow times were accurate to ± 0.1 s, and the uncertainty in the viscosity measurements is 2×10^{-4} mPa · s.

Speeds of sound were determined by a multifrequency ultrasonic interferometer (Mittal Enterprise, New Delhi) operating at 5 MHz, calibrated with water, methanol, and benzene at 298.15 K [11, 15]. The uncertainty of the ultrasonic speed measurements is $0.2 \text{ m} \cdot \text{s}^{-1}$. The details of the methods and techniques have been described elsewhere [2, 11, 14, 15].

3 Results and Discussion

The experimental densities ρ , viscosities η , and excess molar volumes V^E , for the binary mixtures studied at 298.15 K are reported in Tables 2 and 3.

3.1 Excess Molar Volumes

The excess molar volumes V^E , for the mixtures were calculated using the following equation,

$$V^E = \sum_{i=1}^2 x_i M_i \left(\frac{1}{\rho} - \frac{1}{\rho_i} \right) \quad (1)$$

where ρ is the density of the mixture and M_i , x_i , ρ_i are the molar mass, mole fraction, and viscosity of the i th component in the mixture, respectively. The estimated uncertainties for excess molar volumes, V^E , are $0.005 \text{ cm}^3 \cdot \text{mol}^{-1}$.

Figure 1 shows that the excess molar volumes, V^E , for all the cyclohexylamine systems, except for the system involving butylacetate, are negative over the entire range of composition. The strength of interactions of the mixture is in the order: cyclohexylamine + butylamine > cyclohexylamine + *tert*-butylamine > cyclohexylamine + 2-butanone > cyclohexylamine + 2-butoxyethanol > cyclohexylamine + butylacetate.

Table 2 Value of density ρ , viscosity η , excess molar volume V^E , ultrasonic speeds u , and Grunberg–Nissan interaction parameter d_{12} for binary mixtures of cyclohexylamine with butyl acetate, butanone, butylamine, *tert*-butylamine, and 2-butoxyethanol at 298.15 K

x_1	$\rho \times 10^{-3}$ ($\text{kg} \cdot \text{m}^{-3}$)	η (mPa · s)	$V^E \times 10^6$ ($\text{m}^3 \cdot \text{mol}^{-1}$)	u ($\text{m} \cdot \text{s}^{-1}$)	d_{12}
Cyclohexylamine + butyl acetate					
0.0000	0.8744	0.6680	0.000	1176.0	0.0000
0.1151	0.8728	0.6149	0.060	1183.5	-1.9027
0.2265	0.8710	0.6836	0.140	1191.5	-1.1152
0.3342	0.8692	0.8301	0.220	1202.6	-0.4727
0.4385	0.8674	1.0324	0.300	1217.0	0.0502
0.5394	0.8658	1.2533	0.350	1233.6	0.4379
0.6373	0.8643	1.4554	0.390	1255.5	0.7092
0.7321	0.8633	1.6184	0.360	1283.6	0.9106
0.8241	0.8625	1.7342	0.301	1317.2	1.0965
0.9134	0.8621	1.7794	0.186	1362.1	1.2451
1.0000	0.8623	1.7530	0.000	1416.4	0.0000
Cyclohexylamine + butanone					
0.0000	0.7981	0.4000	0.000	1195.0	0.0000
0.0747	0.8053	0.5316	-0.291	1204.3	2.5165
0.1538	0.8125	0.6621	-0.517	1213.8	2.1259
0.2376	0.8198	0.8418	-0.779	1224.9	2.1701
0.3265	0.8272	1.0424	-0.931	1238.4	2.1622
0.4210	0.8339	1.2347	-1.050	1253.3	2.0720
0.5217	0.8407	1.4488	-1.098	1271.9	2.0687
0.6291	0.8471	1.6254	-1.051	1293.9	2.0248
0.7441	0.8529	1.7808	-0.857	1324.9	2.0684
0.8674	0.8581	1.8227	-0.503	1362.3	2.0427
1.0000	0.8623	1.7530	0.000	1416.4	0.0000
Cyclohexylamine + butylamine					
0.0000	0.7319	0.4930	0.000	1251.6	0.0000
0.0757	0.7453	0.6805	-0.291	1258.0	3.2327
0.1557	0.7586	0.8796	-0.517	1266.5	2.9028
0.2401	0.7725	1.1106	-0.779	1276.5	2.7811
0.3296	0.7860	1.3304	-0.931	1288.8	2.6005
0.4244	0.7997	1.5481	-1.050	1302.2	2.4800
0.5252	0.8132	1.7738	-1.098	1318.7	2.4626
0.6324	0.8265	1.9539	-1.051	1337.6	2.4725
0.7468	0.8391	2.0346	-0.857	1358.8	2.4865
0.8691	0.8510	1.9760	-0.503	1384.6	2.5121
1.0000	0.8623	1.7530	0.000	1416.4	0.0000
Cyclohexylamine + <i>tert</i> -butylamine					
0.0000	0.6960	0.4394	0.000	1260.0	0.0000
0.0757	0.7109	0.5690	-0.185	1264.8	2.1951
0.1557	0.7263	0.7445	-0.347	1270.2	2.3730
0.2401	0.7423	0.9554	-0.508	1276.6	2.4353
0.3296	0.7590	1.1727	-0.694	1284.1	2.3785
0.4244	0.7762	1.4070	-0.829	1293.7	2.3601
0.5252	0.7936	1.6123	-0.907	1306.5	2.2990
0.6324	0.8111	1.7854	-0.885	1323.5	2.2666
0.7468	0.8286	1.8944	-0.765	1346.1	2.2631
0.8691	0.8458	1.9153	-0.482	1376.8	2.3702
1.0000	0.8623	1.7530	0.000	1416.4	0.0000

Table 2 continued

x_1	$\rho \times 10^{-3}$ ($\text{kg} \cdot \text{m}^{-3}$)	η (mPa · s)	$V^E \times 10^6$ ($\text{m}^3 \cdot \text{mol}^{-1}$)	u ($\text{m} \cdot \text{s}^{-1}$)	d_{12}
Cyclohexylamine +2-butoxyethanol					
0.0000	0.9020	2.7820	0.000	1305.6	0.0000
0.1169	0.8988	2.8722	-0.130	1302.4	0.8320
0.2295	0.8955	3.0062	-0.245	1303.2	1.0378
0.3380	0.8921	3.0592	-0.331	1306.0	1.1221
0.4427	0.8883	3.0686	-0.369	1311.6	1.2261
0.5437	0.8846	3.0115	-0.397	1320.2	1.3317
0.6412	0.8806	2.8725	-0.390	1333.0	1.4264
0.7355	0.8762	2.6972	-0.326	1348.2	1.5868
0.8266	0.8718	2.4466	-0.248	1366.8	1.7667
0.9147	0.8670	2.0897	-0.121	1390.0	1.7468
1.0000	0.8623	1.7530	0.000	1416.4	0.0000

The negative values of the excess molar volume suggest specific interactions [16] between the mixing components in the mixtures, while its positive values suggest dominance of dispersion forces [1,10] between them. The negative V^E values indicate specific interactions such as intermolecular hydrogen bonding between the mixing components and also interstitial accommodation of the mixing components because of the difference in molar volume. The negative V^E values may also be due to the difference in the dielectric constants of the liquid components of the binary mixture [2]. The very large negative values of V^E for the amines may be attributed to the presence of strong intermolecular hydrogen bond interactions between the cyclohexylamine molecules and the butylamine molecules, and this effect decreases as one shifts from primary to tertiary amines [17,18]. This is probably due to the decreasing hydrogen bond interactions ($-\text{H}-\text{N}-$ $-\text{N}-\text{H}-$) between cyclohexylamine and *tert*-butylamine resulting from the steric effect [19]. Infrared, microwave, and calorimetric studies [10,16,17] have shown the presence of mostly 10-membered dimers and linear associates in $\text{R}-\text{O}-\text{C}_2\text{H}_5\text{OH}$ (where $\text{R} = -\text{CH}_3, -\text{C}_2\text{H}_5, -\text{C}_4\text{H}_9$, etc.), and the small negative values of V^E for the 2-butoxyethanols under investigation may be attributed to the presence of weak hydrogen bonds or dispersive forces [1,2] between the mixing liquids. The positive values of V^E for cyclohexylamine + butyl acetate mixture arises from the close molecular sizes [20] of cyclohexylamine and butylacetate.

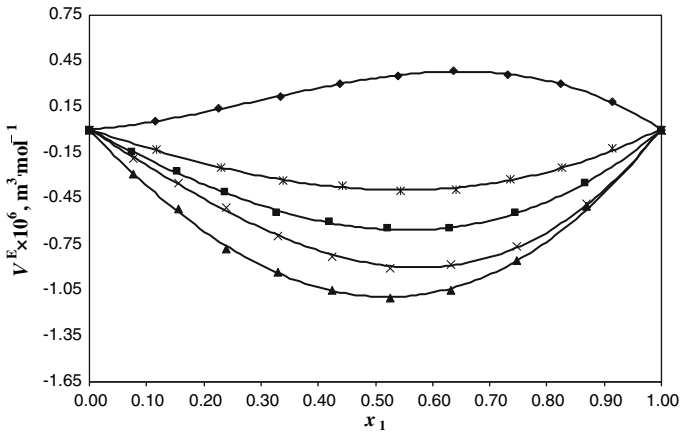
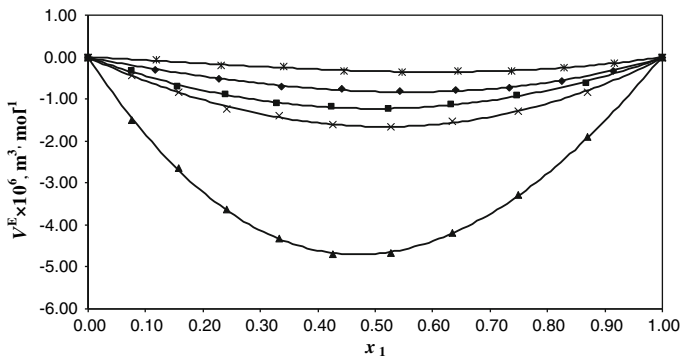
Figure 2 shows that the excess molar volumes, V^E , for all cyclohexanone systems under investigation are negative over the entire range of composition at the experimental temperature. The negative values of excess molar volume for the five systems are in the order: cyclohexanone + butylamine > cyclohexanone + *tert*-butylamine > cyclohexanone + 2-butanone > cyclohexanone + butyl acetate > cyclohexanone + 2-butoxyethanol. The negative values of V^E for the systems cyclohexanone + 2-butanone and cyclohexanone + butyl acetate may be attributed to dipole-induced dipole interactions between the mixing components [21,22] and the small negative values of V^E for the cyclohexanone + 2-butoxyethanol system may be attributed to the presence of weak hydrogen bonds or dispersive forces [1,2] between the mixing liquids.

Table 3 Value of density ρ , viscosity η , excess molar volume V^E , ultrasonic speeds u , and Grunberg–Nissan interaction parameter d_{12} , for the binary mixtures of cyclohexanone with butyl acetate, butanone, butylamine, *tert*-butylamine, and 2-butoxyethanol at 298.15 K

x_1	$\rho \times 10^{-3}$ ($\text{kg} \cdot \text{m}^{-3}$)	η (mPa · s)	$V^E \times 10^6$ ($\text{m}^3 \cdot \text{mol}^{-1}$)	u ($\text{m} \cdot \text{s}^{-1}$)	d_{12}
Cyclohexanone + butyl acetate					
0.0000	0.8744	0.6680	0.000	1176.0	0.0000
0.1162	0.8828	0.6121	-0.310	1173.2	-2.1025
0.2283	0.8907	0.5967	-0.510	1179.3	-2.0747
0.3365	0.8986	0.6230	-0.690	1193.1	-1.9803
0.4410	0.9059	0.6843	-0.767	1212.3	-1.8820
0.5420	0.9131	0.8068	-0.820	1235.5	-1.6556
0.6397	0.9199	0.9528	-0.790	1262.8	-1.5301
0.7341	0.9264	1.1586	-0.730	1293.2	-1.3410
0.8256	0.9322	1.3882	-0.572	1330.1	-1.2647
0.9142	0.9373	1.6710	-0.330	1368.2	-1.2073
1.0000	0.9416	2.0200	0.000	1412.0	0.0000
Cyclohexanone + butanone					
0.0000	0.7981	0.4000	0.000	1195.0	0.0000
0.0755	0.8135	0.4502	-0.341	1193.2	-0.0583
0.1552	0.8294	0.5234	-0.690	1194.0	0.1339
0.2395	0.8443	0.5757	-0.880	1198.3	-0.1296
0.3288	0.8599	0.6546	-1.101	1205.6	-0.1805
0.4235	0.8746	0.7390	-1.171	1219.1	-0.2949
0.5243	0.8896	0.8683	-1.230	1239.5	-0.2963
0.6316	0.9038	1.0302	-1.140	1267.5	-0.3299
0.7461	0.9172	1.2577	-0.911	1305.1	-0.3308
0.8686	0.9306	1.5869	-0.620	1352.7	-0.2505
1.0000	0.9416	2.0200	0.000	1412.0	0.0000
Cyclohexanone + butylamine					
0.0000	0.7319	0.4930	0.000	1251.6	0.0000
0.0765	0.7599	0.6228	-1.490	1256.4	1.7816
0.1570	0.7868	0.7538	-2.659	1264.5	1.5345
0.2421	0.8136	0.9031	-3.640	1271.7	1.4388
0.3319	0.8392	1.0602	-4.320	1281.0	1.3422
0.4270	0.8634	1.2230	-4.690	1291.3	1.2521
0.5278	0.8852	1.3922	-4.670	1304.4	1.1785
0.6349	0.9041	1.5634	-4.201	1319.5	1.1162
0.7488	0.9199	1.7245	-3.290	1341.1	1.0427
0.8702	0.9324	1.8801	-1.890	1369.9	0.9848
1.0000	0.9416	2.0200	0.000	1412.0	0.0000
Cyclohexanone + <i>tert</i> -butylamine					
0.0000	0.6960	0.4394	0.000	1260.0	0.0000
0.0765	0.7176	0.5247	-0.440	1259.5	0.8595
0.1570	0.7402	0.6071	-0.841	1256.6	0.6327
0.2421	0.7640	0.7017	-1.230	1257.4	0.5388
0.3319	0.7876	0.7870	-1.401	1262.6	0.3451
0.4270	0.8128	0.8931	-1.601	1270.6	0.2368
0.5278	0.8384	1.0212	-1.650	1284.8	0.1533
0.6349	0.8642	1.1725	-1.540	1303.9	0.0561
0.7488	0.8906	1.3727	-1.301	1330.6	-0.0164
0.8702	0.9167	1.6443	-0.824	1363.2	-0.0696
1.0000	0.9416	2.0200	0.000	1412.0	0.0000

Table 3 continued

x_1	$\rho \times 10^{-3}$ ($\text{kg} \cdot \text{m}^{-3}$)	η (mPa · s)	$V^E \times 10^6$ ($\text{m}^3 \cdot \text{mol}^{-1}$)	u ($\text{m} \cdot \text{s}^{-1}$)	d_{12}
Cyclohexanone + 2-butoxyethanol					
0.0000	0.9020	2.7820	0.000	1305.6	0.0000
0.1180	0.9063	2.6417	-0.071	1291.6	-0.1344
0.2314	0.9111	2.5177	-0.200	1288.5	-0.1449
0.3404	0.9153	2.4022	-0.230	1292.4	-0.1685
0.4453	0.9199	2.3036	-0.320	1300.9	-0.1869
0.5463	0.9242	2.2167	-0.360	1315.1	-0.2110
0.6436	0.9280	2.1463	-0.331	1332.8	-0.2328
0.7375	0.9321	2.0869	-0.331	1350.2	-0.2656
0.8281	0.9356	2.0478	-0.250	1371.7	-0.2905
0.9155	0.9387	2.0241	-0.141	1392.7	-0.3235
1.0000	0.9416	2.0200	0.000	1412.0	0.0000

**Fig. 1** Variation of excess molar volume, $V^E \times 10^6$ ($\text{m}^3 \cdot \text{mol}^{-1}$), with mole fraction (x_1) of cyclohexylamine at 298.15 K with butyl acetate (\blacklozenge), butanone (\blacksquare), butylamine (\blacktriangle), *tert*-butylamine (\times), and 2-butoxyethanol (\blackstar)**Fig. 2** Variation of excess molar volumes, $V^E \times 10^6$ ($\text{m}^3 \cdot \text{mol}^{-1}$), with mole fraction (x_1) of cyclohexanone at 298.15 K with butyl acetate (\blacklozenge), butanone (\blacksquare), butylamine (\blacktriangle), *tert*-butylamine (\times), and 2-butoxyethanol (\blackstar)

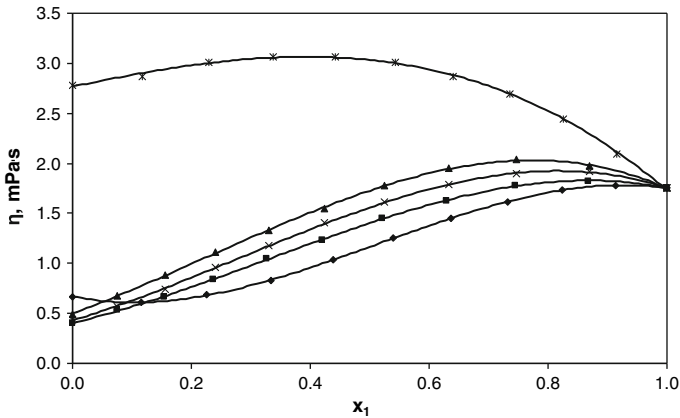


Fig. 3 Variation of viscosity, η (mPa · s), with mole fraction (x_1) of cyclohexylamine at 298.15 K with butyl acetate (◆), butanone (■), butylamine (▲), *tert*-butylamine (×), and 2-butoxyethanol (✱)

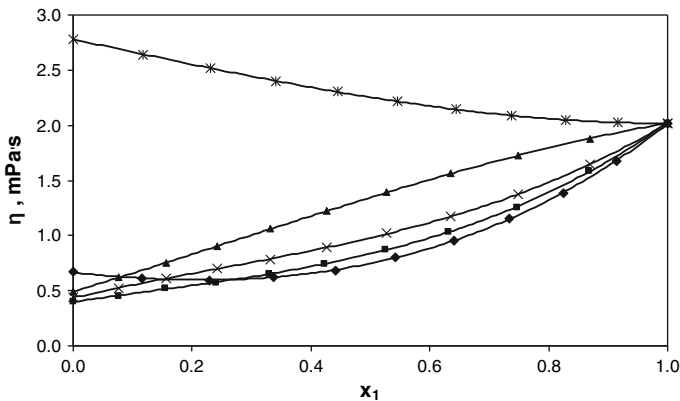


Fig. 4 Variation of viscosity, η (mPa · s), with mole fraction (x_1) of cyclohexanone at 298.15 K with butyl acetate (◆), butanone (■), butylamine (▲), *tert*-butylamine (×), and 2-butoxyethanol (✱)

3.2 Viscosity

From Figs. 3 and 4, the viscosity, η , is seen to increase non-linearly with the concentration of either cyclohexylamine and cyclohexanone for all binaries except for 2-butoxyethanol. The increasing values are due to strong interactions, which suggest specific interactions [16] between the mixing components; whereas, decreasing values are for weak interactions and suggest dominance of dispersion forces [1, 10] between them.

3.3 Viscosity Models and Interaction Parameters

Several semiempirical models have been proposed to estimate the dynamic viscosity (η) of the binary liquid mixtures in terms of pure-component data [23, 24]. One of them we examined is as follows: Grunberg and Nissan [25] have suggested the

following logarithmic relation between the viscosity of the binary mixtures and the pure components, where d_{12} is a constant proportional to the interchange energy:

$$\eta = \exp \left[\sum_{i=1}^j (x_i \ln \eta_i) + d_{12} \prod_{i=1}^j x_i \right]. \tag{2}$$

It may be regarded as an approximate measure of the strength of molecular interactions between the mixture components. The values of the interchange parameter d_{12} have been calculated as a function of the composition of the binary liquid mixtures of cyclohexylamine and cyclohexanone with butyl acetate, butanone, butylamine, *tert*-butylamine, and 2-butoxyethanol as listed in Tables 2 and 3. It has been found that the values of d_{12} are positive for all the cyclohexylamine binary systems studied, except

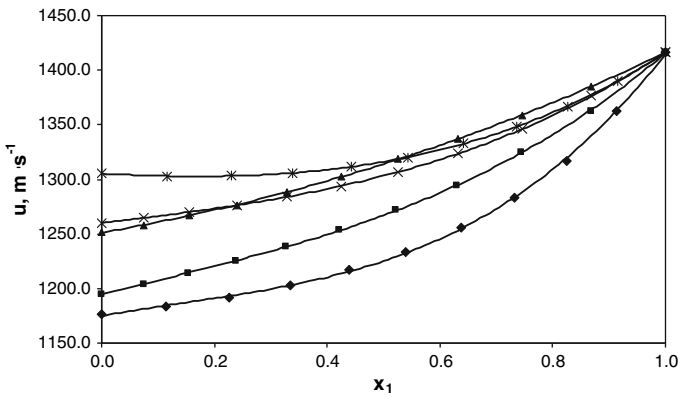


Fig. 5 Variation of ultrasonic speed, u ($\text{m} \cdot \text{s}^{-1}$), with mole fraction (x_1) of cyclohexylamine at 298.15 K with butyl acetate (◆), butanone (■), butylamine (▲), *tert*-butylamine (×), and 2-butoxyethanol (✱)

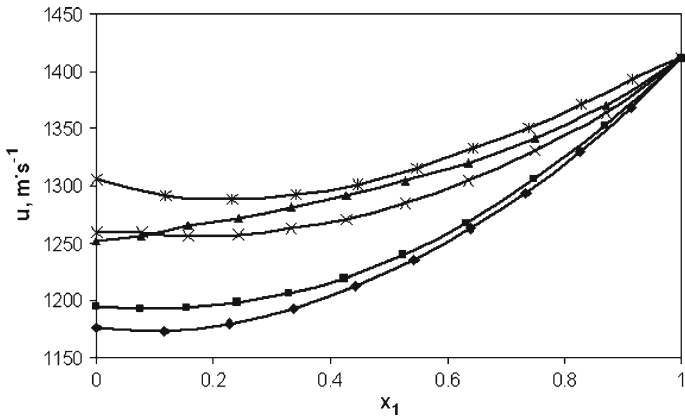


Fig. 6 Variation of ultrasonic speed, u ($\text{m} \cdot \text{s}^{-1}$), with mole fraction (x_1) of cyclohexanone at 298.15 K with butyl acetate (◆), butanone (■), butylamine (▲), *tert*-butylamine (×), and 2-butoxyethanol (✱)

for the systems involving cyclohexanone + (butylacetate, butanone, and 2-butoxyethanol). The negative values of d_{12} indicate the presence of dispersion forces [26] between the components in the mixtures, while its positive values indicate the presence of specific interactions [26] between them.

3.4 Speeds of Sound

In Figs. 5 and 6, the speeds of sound, u , have been plotted against the mole fraction of cyclohexylamine or cyclohexanone at 298.15 K. The increasing trends are justified by the presence of weak interactions [1, 10] or structural-disruptive effects between the mixing liquids for the binary mixtures of alkoxyethanol and by the presence of strong hydrogen bond interactions [16] between the mixing liquids for the binary mixtures of amines.

4 Conclusions

In summary, amine systems are characterized by the presence of strong hydrogen bond interactions between the mixing liquids and the strength of interactions follows the order: primary amine > tertiary amine; also steric and other effects play a pivotal role in this regard. On the contrary, 2-butanone systems are expressed by hydrogen bonding as well as dipole–dipole interactions resulting in a charge transfer complex, while alkoxyethanols systems are characterized by the presence of weak hydrogen bond interactions or dispersive forces in the studied binary systems. The reason for these types of behaviors is probably the formation of intramolecular associates in these molecules by the interaction of the etheric oxygen and hydrogen of the –OH group in the same alkoxyethanol molecule.

Acknowledgment The authors are grateful to the Departmental Special Assistance Scheme, Department of Chemistry, N.B.U. under the University Grants Commission, New Delhi (No. F540/27/DRS/2007, SAP-1), for financial support in connection with this research work.

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